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7-02 06-07 DE 1904506-19N/08/13 CORG 63/16, A61K 7/03, C08G 63/16, A61K 7/03, C08G 63/06, C09D 163/00

Chiral nematic polyester with stable Grandjean texture - contains iso-sorbide, iso-mannide and/or iso-lidide units and specified chain-flexibilising units derived from flexible diacid and diol or diphenol.

98-133293

Addin Data KRICHELDORF H R, KRAWINKEL T, GERKEN A,

SCHUHMACHER P

Chiral nematic polyesters with flexible chains (D), containing iso-sorbide, iso-mannide and/or iso-lidide units and at least one flexibilising unit selected (and derived) from (a) aliphatic dicarboxylic acids, (b) aromatic dicarboxylic acids with flexible spacers, (c) *o*-alkanediols, (d) di phenols with flexible spacers and (e) condensation products of poly(ethylene terephthalate)s or naphthalates with acylated diphenols and acylated iso-sorbides.

Also claimed are (i) pigments containing (D), (ii) coating materials containing (D) or pigments as in (ii), (iii) processes for the production of (D)

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USE

As optical components, coating materials, colouring agents especially colour components in paint systems for coating surfaces and components of printing ink, and in cosmetic materials, especially nail varnish and lipstick (claimed).

ADVANTAGE

Provides chiral nematic polyesters which form stable Grandjean textures without showing an increased tendency to crystallisation. These polyesters are made by a simple, low-cost process and fixed without crosslinking by cooling to below the glass transition temperature (T_g), which is generally above 80 (preferably above 100) $^{\circ}\text{C}$ in spite of flexibilisation.

CLAIMED PROCESS

The production of (D) comprises (A) reacting free diols with dicarboxylic acid chlorides in inert aromatic solvent, especially 1-chloronaphthalene, or (B) reacting silylated diols with diacid dichlorides in bulk, or (C) reacting acetylated diols with free diacids,

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(or (D)) reacting a mixture of free diacids and acetylated diols with a polyester, especially polyethylene or polybutylene terephthalate or polyethylene naphthalate.

PREFERRED COMPOSITION

Preferred flexibilising units are units of formula (a) -CO-(CH₂)_n-CO₂, especially adipic acid units, (b) -CO-Phe-A-Phe-CO-, (c) -(CH₂)_mO- or -O-(CH₂)_nO- and (d) -O-Phe-A-Phe-O-,

Phe = 1,4-phenylene;

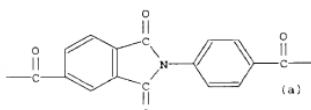
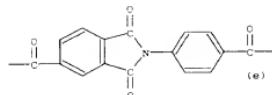
A = -CO-(CH₂)_p-O- or -(CH₂)_p-O-(CH₂)_q-;

n = 3-15, preferably 4-10;

m = 1-10;

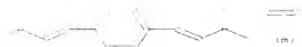
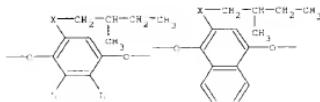
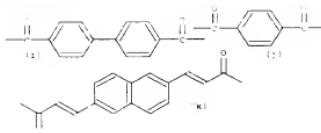
n, p = 1-7.

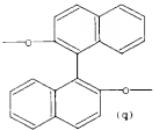
Non-flexible acid components comprise units of formulae (e) - (iii). Non flexible alcohol components comprise units of formulae (ii) - (iii).



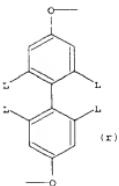
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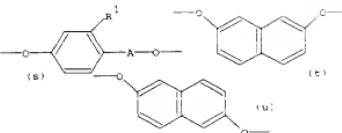




(q)



(r)



L = alkyl, alkoxy, halogen, COOR, OCOR, CONHR or NHCOR;
 X = S, O, N, CH- or a single bond;
 A = single bond, -CH=, Cyc-, -CH-CH-, Cyc-, -Cyc-, -X-Phe- or 2,6-naphthylene;
 Cyc = 1,4-cyclohexylene;
 Phe = 1,4-phenylene;
 R' = H, halogen, alkyl or phenyl;
 R = H or alkyl.

(II) contain 1-15 (2.5-10) mol% isosorbide, isomannide and/or isocitride units and 1-50 (5-30) mol% chain-flexibilising units. (I) show an

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inherent viscosity of 0.1-3 (0.1-1.5) dL/g at 20°C and a Tg of 80-300 (100-220)°C.

EXAMPLE

A mixture of 19 mmols tert-butylhydroquinone, 1 mmol isosorbide, 2 mmols adipoyl chloride, 18 mmols N-(4'-carboxyphenyl)-trimellitic acid imide chloride and 20 ml 1-chloronaphthalene was heated rapidly to 230°C and then stirred for 8 hrs. at this temperature under nitrogen. The mixture was worked up by dissolution in dichloromethane/trifluoroacetic acid (DCM/TFA; 4:1), precipitation in methanol and drying under vacuum at 80°C, to give polyester (I-1a) in 98% yield. The product had an inherent viscosity (in 4/1 DCM/TFA at 20°C) of 0.57 dL/g, a Tg of 195°C and a clearing pt. of above 400°C. It had a blue colour and showed a Grandjean texture. (PW)

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